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Short Communication

Development and characterization of laser clad high temperature self-lubricating wear resistant composite coatings on Ti–6Al–4V alloy

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ABSTRACT

To enhance the wear resistance and friction-reducing capability of titanium alloy, a process of laser cladding γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS coatings on Ti–6Al–4V alloy substrate with preplaced NiCr/Cr_3C_2–WS_2 mixed powders was studied. A novel coating without cracks and few pores was obtained in a proper laser processing. The composition and microstructure of the fabricated coating were examined by X-ray diffraction (XRD), scanning electron microscope (SEM), energy dispersive spectroscopy (EDS) techniques, and tribological properties were evaluated using a ball-on-disc tribometer under dry sliding wear test conditions at 20 °C (room-temperature), 300 °C, 600 °C, respectively. The results show that the coating has unique microstructure consisting of α-Ti, TiC, $TiWC_2$, γ-NiCrAlTi, Ti_2CS and CrS phases. Average microhardness of the composite coating is 1005 $HV_{0.2}$, which is about 3-factor higher than that of Ti–6Al–4V substrate (360 $HV_{0.2}$). The friction coefficient and wear rate of the coating are greatly decreased due to the combined effects of the dominating anti-wear capabilities of reinforced TiC and $TiWC_2$ carbides and the CrS and Ti_2CS sulfides which have excellent self-lubricating property.

1. Introduction

Titanium alloys are widely used for various industrial fields, such as aerospace engineering, biomedical and power generation due to their several property advantages, such as high strength to weight ratio, low density, low modulus, excellent corrosion resistance and biocompatibility [1–3]. But their utility is also restricted by the low surface hardness (360 HV) and poor tribological properties (high and unstable friction coefficient, low high-temperature abrasive and adhesive wear resistance) [4,5], especially for the application of key moving components. Therefore, there is an increasing interest in improving surface properties of titanium alloy through various surface modification techniques, such as plasma spraying [6], physical vapor deposition [7], chemical vapor deposition [8], and nitriding [9,10]. The development of these techniques makes a great advance, whereas they give rise to many difficulties, such as poor adherence, lower bonding strength, some defects at the interface. More recently, laser cladding technology is a promising way to overcome the above difficulties with many advanced features, such as metallurgical bond between the coating and substrate, high refined microstructure, low dilution ratio and limited heat affected zone [11–13].

With the aforementioned advantages, laser cladding is a dominating and popular surface technology to fabricate wear-resistant coating on titanium alloy. Yang et al. [14] synthesized Ti–Si–C–N/TiCN composite coating on Ti–6Al–4V by laser cladding. It was found that the microhardness of the coating was about 1400 HV, which was about 4 times higher than that of titanium substrate. Li et al. [15] fabricated Ti_3Al/TiAl + TiC ceramic layer deposited by laser cladding on Ti–6Al–4V alloy. The result showed that the wear resistance of the composite layer was approximately 2 times greater than that of the titanium substrate due to the reinforcement of the Ti_3Al/TiAl + TiC hard phase. Although the above mentioned coatings are beneficial to improve the tribological property of the titanium alloy with the increasing of surface hardness themselves, they usually present high friction coefficient under friction conditions of high contact stress, high temperature and no liquid lubrication, which is harmful to the durability of titanium alloy, especially for the tribological counterpart component [16]. One of the feasible methods to reduce friction coefficients is to add solid lubricant particles such as MoS_2, CaF_2 and graphite to the composite coating [17,18].

As a well known solid lubricant, WS_2 has lamellar structure with low shear strength like MoS_2 and graphite, and it is easy to form transfer lubricious films on the tribological contact of working surface. In addition, WS_2 has relative high oxidation temperature (539 °C) than MoS_2 (370 °C) or graphite (325 °C), so it can

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maintain lubricating property at relative higher temperature [19]. In our previous study, the efforts of fabricating high temperature self-lubricant wear-resistant composite coatings on austenitic stainless steel with different constitution of NiCr–Cr_3C_2–WS_2 [20] and Ni80Cr20–Cr_3C_2 [21] precursor mixed powders were made, the results showed that the composite coatings could reduce the friction coefficient and wear loss of the substrate in a great deal. To the best knowledge of the authors, only very few literatures are available concerned on the synthesis of the above similar self-lubrication wear resistant composite coating on the Ti–6Al–4V alloy by laser cladding.

In this work, the high temperature self-lubricating wear resistant composite coating was designed and fabricated with NiCr/Cr_3C_2 and WS_2 mixed powders on Ti–6Al–4V substrate by laser cladding. The microstructure, microhardness and tribological properties of the composite coatings were investigated systemically. The aim is to provide a novel way for synthesizing the high temperature self-lubricant wear resistant composite coatings on titanium alloy and promote its commercial application.

2. Experimental procedures

2.1. Material

The substrate material used in the experiment was Ti–6Al–4V alloy, the chemical compositions of the Ti–6Al–4V alloy were shown in Table 1. Specimens of 50 mm × 40 mm × 8 mm were cut from the cast and homogenized ingots by electric discharge wire cutting machine, and the 50 mm × 40 mm surface as the cladding surface. Before laser cladding, the working surface of the specimens was ground to a surface roughness of R_a = 0.2 μm, and rinsed with ethanol and acetone. The mixtures of NiCr/Cr_3C_2–30%WS_2 (wt.%) were mixed by using a QM-ISP04 series ball miller and used as the precursor preplaced material. The composite powders were then mixed with cellulose acetate, pasted onto the substrate and dried in an oven at 80 °C for 2 h. The thickness of the layers was approximate 1.0–1.5 mm.

2.2. Cladding system and parameters

The laser cladding experiments were carried out in a laser processing system (GS-TFL-10 kW, China) consisting of a 10 kW transverse-flow continuous-wave CO_2 laser with a computer-controlled multi-axis positioning workstation. The final laser cladding parameters were: laser output power 1.5 kW, beam diameter 4.0 mm, beam traverse speed 6 mm/s. N_2 shielding gas was used to protect the melt pool from oxidation.

2.3. Characterization of coating and tests

After laser cladding treatment, metallographic samples and wear testing specimens were cut by electro-sparking machining, all the clad samples were first resin mounted, polished and etched with HF:HNO_3:H_2O water solution in volume ratio of 1:3:9, and the samples for wear testing were then surface smoothed by mechanical milling and grinding to acquire the surface roughness of R_a = 0.8 μm. X-ray diffraction (XRD) was characterized using a Pert-Pro MPD X-ray diffractometer with Cu target Kα radiation to identify the phase constitutions. The microstructures of the cross section of layer and substrate-coating interface were performed employing a KYKY-EM3200 S-4700 scanning electron microscope (SEM, Japan) equipped with an energy dispersive X-ray system (EDS).

MH-5 type Vickers microhardness was measured with a 200 g load and dwell time of 15 s on sections perpendicular to the direction from the coating surface to the substrate. At least 10 random indentation measurements were averaged for each sample. The friction and wear properties of the coatings and substrate were tested on a ball-on-disk tribo-meter (HT-1000 tester, Lanzhou Zhongkekaihua science and technology Co., Ltd., China) under dry sliding condition at ambient (20 °C), 300 °C, 600 °C environment against a Φ 4 mm Si_3N_4 ceramic ball with a hardness of 1700 HV respectively, and the test parameters were listed in Table 2. The wear volume was measured with a surface contour displacement transducer. The wear rate was calculated with the formula below:

$$W = \frac{V}{LS} \tag{1}$$

where W is wear rate; V is wear volume (mm^3); L is load (N); S is the total sliding distance (m).

Table 1
Chemical compositions of the Ti–6Al–4V alloy (wt.%).

Ti	Al	V	Fe	C	N	O	Others
Bal.	6.3	4.2	0.11	0.03	0.03	0.15	⩽0.40

Table 2
Experimental parameters of wear test.

Load/N	Temperature/°C	Wear time/min	Rotation radius/mm	Linear velocity/($m\ min^{-1}$)
5	20,300,600	40	2	16.89

3. Results

3.1. Microstructure

Fig. 1(a) shows the micrographs of the overview cross-section of the laser cladding γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS composite layer with few holes and cracks. The bonding of the coating to the substrate is completely metallurgical, as clearly demonstrated in Fig. 1(b). It is found that the microstructure of the bottom is different. Due to the high temperature gradient and non-equilibrium solidification at the bottom of the molten pool, the epitaxial growth of columnar crystal from the substrate can be seen clearly. While the cell, irregular grains present in the region which have a distance from the fusion line about 25 μm (region A), it is because that the grain remains un-melted or primary solidified phase in the molten pool can form the new grain nucleation core at the interface between solid and liquid due to the intense convection in molten pool, and the newly formed grains grow up with cellular morphology [22].

X-ray diffraction performed on the clad sample shows that the major phases of the cladding layer are α-Ti, TiC, γ-NiCrAlTi, and $TiWC_2$. Despite their low intensity diffraction peaks, Ti_2CS and CrS phases are also unambiguously identified, since all strong diffraction peaks of this constituent are present in the diffraction diagram, as can be seen from Fig. 2. Due to the very high energy density of the laser beam irradiation, most WS_2 powder decomposed to S element, which reacted with the melting Cr and also integrated with the primary TiC to transform to CrS and Ti_2CS sulfides [19,23].

Fig. 3 is the typical microstructure of the laser clad composite coating with high magnification. Through careful examination, it shows that there are mainly four phases existing in the coating. The dark gray irregular structure marked as region A, which are enriched in Ti and C by EDS analysis (see Table 3), while area B are gray blocky structure and its composition is W, C and Ti, and their content is relatively even. The white light flocculent and marked as arrow C, which is enriched in Ni, Cr, Al and Ti. Finally, the isolated

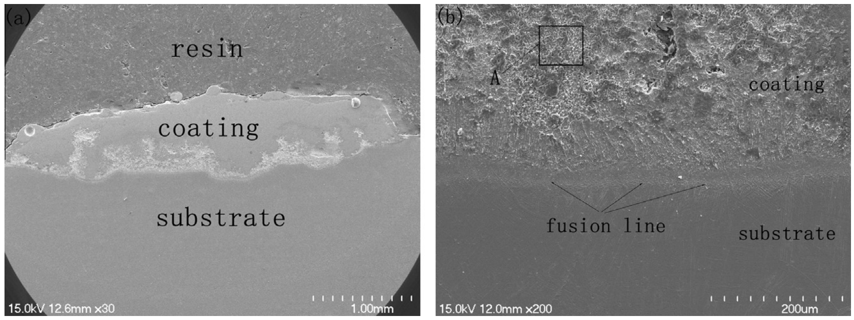

Fig. 1. SEM photographs of the laser clad γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS composite coating. (a) Cross-section overview and (b) intermediate region.

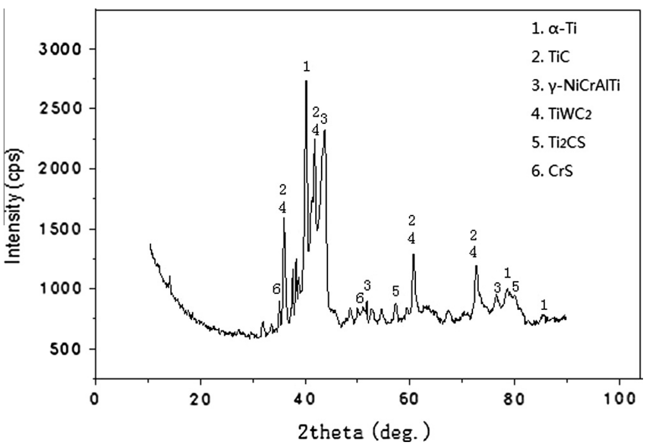

Fig. 2. XRD pattern of the laser clad γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS composite coating.

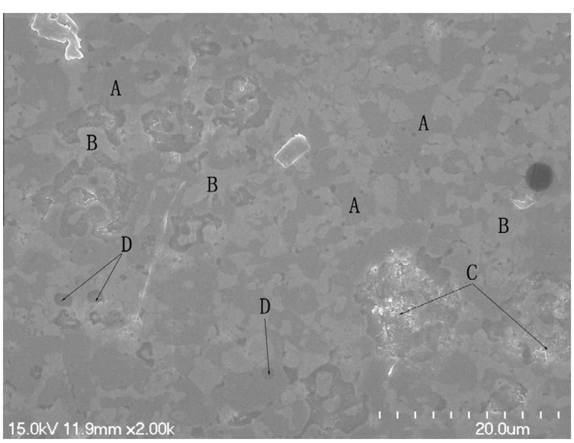

Fig. 3. Typical microstructure of the laser clad γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS composite coating.

and fine spherical particles dispersed in the matrix, and marked as arrows D, consists of S, Cr, C, and Ti, also including W and Ni. According to the XRD results, it is believed that the regions A and B are TiC and $TiWC_2$ carbides, respectively, which contribute to the hardness and wear resistance of the coating, while the region C is mainly γ-NiCrAlTi solid solution. Finally, it can be inferred that CrS and Ti_2CS sulfides are agglomerate in the area D.

3.2. Microhardness

Fig. 4 presents the microhardness depth profile of the transverse cross-section of laser clad composite coating. It is found that

Table 3
Compositional analysis of laser clad composite coating on Ti–6Al–4V alloy.

Area	Composition (wt.%)							
	Ni	Cr	C	W	S	Ti	Al	V
A	1.46	1.97	18.63	2.88	–	74.50	0.56	–
B	2.58	3.30	13.84	60.30	0.71	19.27	–	–
C	7.39	27.67	3.60	2.96	–	51.71	4.09	2.58
D	5.20	32.91	15.47	3.65	25.48	17.29	–	–

the microhardness fluctuates in the range of 552–1300 $HV_{0.2}$ in the coating zone, and its average microhardness is 1005 $HV_{0.2}$, which is about more than 3-factor improvement to that of Ti–6Al–4V substrate (360 $HV_{0.2}$). It is because that large amount of reinforced TiC and $TiWC_2$ carbides are uniformly distributed in the coating, the laser induced fine microstructure is also beneficial to the improvement of microhardness due to microstructure refining strengthening, as can be seen in the micrographs.

3.3. Tribological performance

The friction coefficients of the γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS composite coating and the substrate as the function of temperature are shown in Fig. 5. It could be found that with an increase in test temperature, the friction coefficient of the laser clad coating decreases firstly, then increases slightly and finally fluctuates about at 0.316 (20 °C), 0.301 (300 °C) and 0.336 (600 °C), respectively, while the friction coefficient of titanium alloy reduces from 0.489 to 0.419 gradually. That is to say, the friction coefficients of

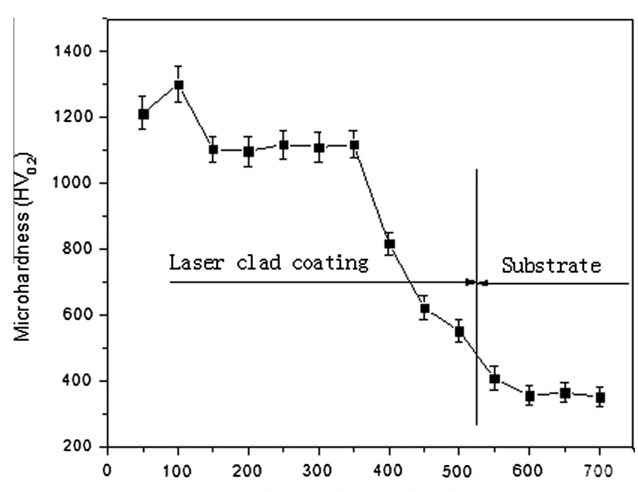

Fig. 4. Microhardness distribution of the laser clad composite coating.

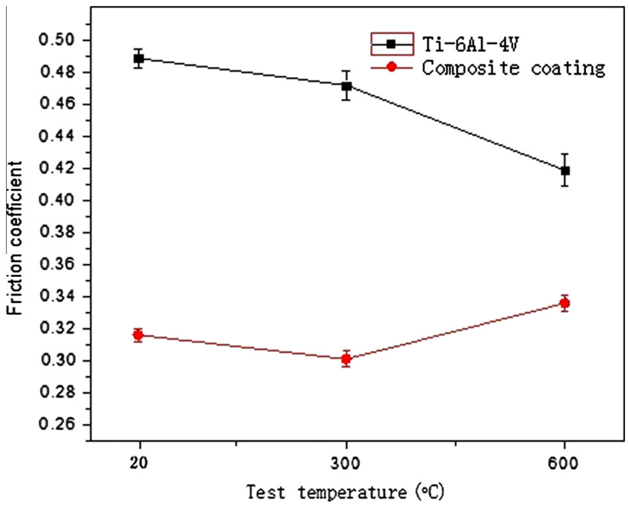

Fig. 5. Friction coefficient of Ti–6Al–4V substrate and laser clad coating as functions of temperature.

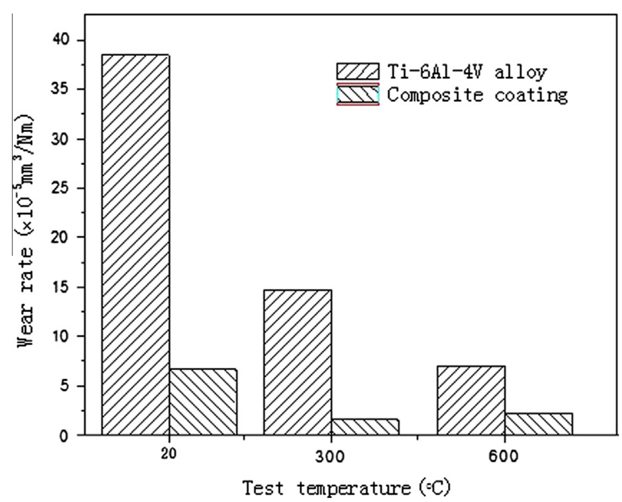

Fig. 6. Wear rate of the Ti–6Al–4V substrate and laser clad coating as functions of temperature.

the composite coating are reduced by 19.8–36.2% compared with those of the titanium substrate under different temperatures. Further, the wear rates of the composite coating and the substrate are shown in Fig. 6, as we can see, all of the wear rates of the composite coating are great lower than those of the Ti–6Al–4V alloy obviously at different test temperatures, and the wear rates of the composite coating are 68.9–89.5% less than those of the substrate. Especially, the composite coating exhibits excellent wear resistance at room and moderate temperature. Thus, the γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS composite coating has distinguished friction-reducing and anti-wear abilities due to the synergetic effects of very hard TiC, $TiWC_2$ reinforced carbides and relatively soft CrS, Ti_2CS self-lubricating sulfides which have low shear strength.

4. Discussion

In order to understand the underlying wear mechanisms of the γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS high temperature self-lubricating composite coating, SEM images and EDS analysis of the worn surfaces of the clad coating and the substrate were conducted. Fig. 7 shows the typical morphology of the worn surfaces of the Ti–6Al–4V alloy substrate and the laser clad γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS composite coating at different test temperatures. The morphologies at different temperatures are similar for Ti–6Al–4V substrate, demonstrating that the wear mechanism with an increase of the temperature is similar, while it is obviously different for the laser clad composite coating. It can be seen from Fig. 7(a,c,e) that the Ti–6Al–4V substrate suffered severe abrasive wear evidenced by serious plastic deformation, break off and deep plowing grooves. It is because that the asperities of the very hard Si_3N_4 ceramic ball counterpart as grinding grains can be pressed into the softer surface of Ti–6Al–4V alloy with the continuous sliding friction proceeding. In addition, the microgrooves of the worn Ti–6Al–4V alloy are more obvious, and the plastic deformation is relatively more mild at high temperature, especially at 600 °C with some careful observation. According to the EDS analysis results of the worn surface of the Ti–6Al–4V substrate at 600 °C (Table 4), it can be seen that the oxygen contents of the worn titanium alloy surface are relatively very high, indicating oxidation of the frictional surface and the formation of the softer oxidation films which have low shearing strength and good toughness. Similar phenomenon was also observed by Cheng et al. [24], the wear resistance of the Ti–46Al–2Cr–Nb intermetallics was significantly enhanced due to the occurrence of brittle to ductile transition and the formation of thick protective oxide layer on the worn surfaces at 750 °C. With the formation of oxidation films, Ti–6Al–4V alloy showed improved wear resistant property under high temperature (600 °C), which was in accordance with the variation tendency of the friction coefficient and the wear rate of Ti–6Al–4V substrate as the function of the test temperature. The wear mechanisms of Ti–6Al–4V substrate are mainly abrasive wear, micro-plough and plastic deformation [25,26].

On the other hand, the worn surfaces of the γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS composite coating are much smoother than that of titanium alloy, with un-conspicuous plastic deformation and micro-ploughing, as shown in Fig. 7(b,d,f). The difference in the wear behavior is due to the existence of reinforced TiC and $TiWC_2$ carbides and lubricous CrS, Ti_2CS sulfides in the coating as compared to the substrate, which contributes to its excellent resistance to plastic deformation and scraping by the counterpart and other wear debris. Fig. 7(b) shows the morphology of the worn surface of composite coating at room temperature (20 °C), it is found that many microscopic pits are on the surface. It may be because that part of the hard phases pressed into the tough matrix under the sustained normal load and contact stress, and the other were ground off by Si_3N_4 ceramic counterpart as wear debris to promote three-body abrasive wear between the sliding surfaces. In addition, the ductile and tough γ-NiCrAlTi eutectic matrix could play the very important role of firmly connecting and supporting the hard phases and preventing the reinforced wear resistant phases from peeling on the worn surface, which is favorable to reduce the wear loss of the coating [27]. The abrasive and mild adhesive wear is the main wear mechanism of the composite coating at room temperature.

As shown in Fig. 7(d), the worn surface of the composite coating at 300 °C is also very smooth except some relative big pits in comparison with room temperature (20 °C), and covered with some transferred layers. It indicates that the CrS and Ti_2CS are sheared to form transfer films at the contact interface due to their inherent soft and solid lubricant character (i.e. it prevents adhesion and has special microstructure with low shear strength) [19,23]. Moreover, the soft transferred layer made the friction and wear between Si_3N_4 ceramic ball and the composite coating change into that between the former and the transferred layer. So the friction coefficients and the wear rates of the composite coating are greatly reduced because of the solid lubrication effect of the transferred layer. Through some careful observation, slight plastic deformation features present on the uncovered surface. Therefore, the wear mechanism of the composite coating is mainly the generation of the transfer film.

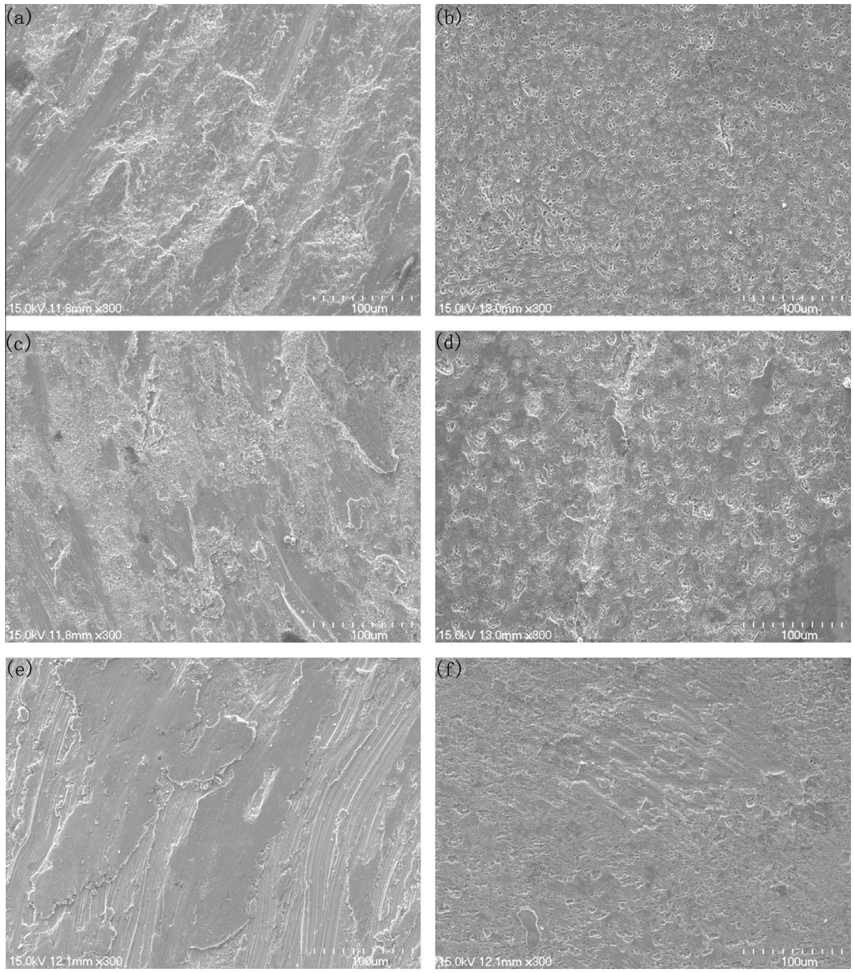

Fig. 7. Wear morphologies of Ti–6Al–4V substrate at (a) 20 °C, (c) 300 °C, (e) 600 °C and γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS composite coating at (b) 20 °C, (d) 300 °C, (f) 600 °C.

Table 4
EDS analysis results of the worn surface of Ti–6Al–4V alloy and laser clad coating after the dry sliding wear test at 600 °C.

Sample	Ti	Ni	Al	Cr	C	W	S	O
Ti–6Al–4V	72.9	–	4.06	–	–	–	–	23.04
Laser clad coating	21.11	8.61	–	20.64	4.22	15.48	2.75	27.19

However, partial peeling and mild grooves are present in the worn surface at 600 °C (Fig. 7(f)) because of the continuous sliding friction, the decomposition and oxidation of the CrS, and Ti_2CS sulfides. At the same time, the resistance to shearing would be weakened due to the delamination of the transferred layer. Thus, some mild grooves along the sliding direction present on the worn surface. In addition, the existence of γ-NiCrAlTi eutectic matrix having excellent properties of ductility and toughness plays a dominate role in preventing very hard TiC and $TiWC_2$ ceramic particles from removal by toughening them and relieving the stress concentration, while they may be softened at high temperature (600 °C) and make the wear debris easily adhere to the surface and promote three-body abrasive wear, which is similar to the results of Feng et al. [28]. According to the EDS analysis results (Table 4), it can be seen that the oxygen contents of the worn surface are relatively very high, indicating obvious oxidation of the frictional surface and forming NiO, CrO and Cr_2O_3 at 600 °C [29,30]. So the main wear mechanisms of the composite coating are the breakdown of transfer layer and oxidative wear.

5. Conclusions

The γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS high temperature self-lubricating composite coating on Ti–6Al–4V alloy substrate with preplaced NiCr/Cr_3C_2–WS_2 powders was fabricated by laser cladding. The corresponding processing techniques, microstructure evolution and friction-reducing and anti-wear capabilities as well as the wear mechanisms of the composite coating were studied systemically. Based on the experimental results of the investigation, the following conclusions can be drawn:

(1) The γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS high temperature self-lubricating wear resistant composite coating with few pores and no cracks could be obtained on Ti–6Al–4V substrate by laser cladding technology.
(2) The microstructure of the composite coating consists of α-Ti, TiC, $TiWC_2$, γ-NiCrAlTi, Ti_2CS and CrS phases. Due to the decomposition of WS_2 powder, sulfur reacted strongly with the melt Cr and the primary TiC dendrites, forming CrS and Ti_2CS sulfides in the molten pool.
(3) The average microhardness of the composite coating is 1005 $HV_{0.2}$, which is about more than 3-factor improvement to that of Ti–6Al–4V substrate (360 $HV_{0.2}$). It is because that

the existence of a large amount of reinforced TiC and $TiWC_2$ carbides homogeneously distributed in the matrix and the refined microstructure of the coating.

(4) The composite coatings showed low friction coefficients and reduced wear rates at room temperature up to 600 °C, the friction coefficients reduced to 0.301 and the wear rates reduced by 89.5% compared to that of Ti–6Al–4V substrate at 300 °C, which are attributed to the combined effects of the dominating anti-wear capabilities of reinforced TiC and $TiWC_2$ carbides and self lubricating properties of Ti_2CS and CrS sulfides. The tribological property of the Ti–6Al–4V alloy substrate was significantly improved after laser clad γ-NiCrAlTi/TiC + $TiWC_2$/CrS + Ti_2CS high temperature self-lubricating wear resistant composite coating.

(5) The wear mechanism of Ti–6Al–4V substrate is mainly abrasive wear and micro-ploughing, while that of the composite coating is the generation and breakdown of transfer layer and oxidative wear.

Acknowledgements

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